
	Massachusetts Department of Environmental Protection Bureau of Waste Site Cleanup	WSC-CAM	Section: V C
		July 1, 2010	Revision No. 1
		Final	Page 1 of 29
Quality Control Requirements and Performance Standards for the <i>Analysis of Chlorinated Herbicides by Gas Chromatography (GC)</i> in Support of Response Actions under the Massachusetts Contingency Plan (MCP)			

WSC – CAM – V C

Quality Control Requirements and Performance Standards for
the ***Analysis of Chlorinated Herbicides by Gas
Chromatography (GC)*** in Support of Response Actions under
the Massachusetts Contingency Plan (MCP)

	Massachusetts Department of Environmental Protection Bureau of Waste Site Cleanup	WSC-CAM	Section: V C
		July 1, 2010	Revision No. 1
		Final	Page 2 of 29
Quality Control Requirements and Performance Standards for the <i>Analysis of Chlorinated Herbicides by Gas Chromatography (GC)</i> in Support of Response Actions under the Massachusetts Contingency Plan (MCP)			

V. Gas Chromatography (GC) Methods

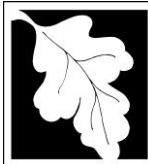
C. Quality Control Requirements and Performance Standards for WSC-CAM-V C (Chlorinated Herbicides by GC)

Table of Contents

Acronym List	3
1.0 Quality Control Requirements and Performance Standards for WSC-CAM-V C	4
1.1 Overview of WSC-CAM-V C	4
1.2 Summary of SW-846 Method 8151A	6
1.3 Sample Extraction/Cleanup Methods for WSC-CAM-V C	7
1.4 Method Interferences	8
1.5 Quality Control Requirements for WSC-CAM-V C	9
1.6 Special Analytical Considerations for WSC-CAM-V C	9
1.7 Analyte List for WSC-CAM-V C	20
2.0 Data Usability Assessment	23
3.0 Reporting Requirements for WSC-CAM-V C	23
3.1 General Reporting Requirements for WSC-CAM-V C	23
3.2 Specific Reporting Requirements for WSC-CAM-V C	23

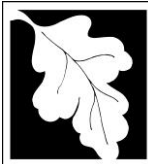
List of Tables and Appendices

Table V C-1	Specific QC Requirements and Performance Standards for WSC-CAM-V C	11-19
Table V C-2	Analyte List for WSC-CAM-V C	22
Table V C-3	Routine Reporting Requirements for WSC-CAM-V C	24
Appendix V C-1	Sample Collection, Preservation and Handling Procedures for Chlorinated Herbicide Analyses	25-26
Appendix V C-2	Data Deliverable Requirements for Data Audits	27-29

	Massachusetts Department of Environmental Protection Bureau of Waste Site Cleanup	WSC-CAM	Section: V C
		July 1, 2010	Revision No. 1
		Final	Page 3 of 29
Quality Control Requirements and Performance Standards for the <i>Analysis of Chlorinated Herbicides by Gas Chromatography (GC)</i> in Support of Response Actions under the Massachusetts Contingency Plan (MCP)			

ACRONYM LIST

CAM	Compendium of Analytical Methods	MS	Matrix spike
CASN	Chemical Abstracts Service Number	MSD	Matrix spike duplicate
CCAL	Continuing calibration	NA	Not applicable
%D	Percent difference or percent drift	PTFE	Polytetrafluoroethylene
2,4-D	2,4-Dichlorophenoxy acetic acid	QA	Quality assurance
2,4-DB	2,4-Dichlorophenoxy butyric acid	QC	Quality control
DBOFB	4,4'-Dibromooctafluorobiphenyl	r	Correlation coefficient
DCAA	2,4-Dichlorophenylacetic acid	r ²	Coefficient of determination
DF	Dilution factor	RAO	Response Action Outcome
ECD	Electron capture detector	RCs	Reportable Concentrations
GC	Gas chromatograph	RL	Reporting limit
ICV	Initial calibration verification	RPD	Relative percent difference
IRAs	Immediate Response Actions	RQs	Reportable Quantities
LCS	Laboratory control sample	%RSD	Percent relative standard deviation
MassDEP	Massachusetts Department of Environmental Protection	2,4,5-T	2,4,5-Trichlorophenoxy acetic acid
		µg/kg	micrograms per kilogram
		µg/L	micrograms per liter
MCP	Massachusetts Contingency Plan		
MCPA	2-Methyl-4-chlorophenoxy acetic acid		
MCPP	2-(2-methyl-4-chlorophenoxy) propionic acid		
MD	Matrix duplicate		
MOHML	Massachusetts Oil and Hazardous Materials List		

	Massachusetts Department of Environmental Protection Bureau of Waste Site Cleanup	WSC-CAM	Section: V C
		July 1, 2010	Revision No. 1
		Final	Page 4 of 29
Quality Control Requirements and Performance Standards for the <i>Analysis of Chlorinated Herbicides by Gas Chromatography (GC)</i> in Support of Response Actions under the Massachusetts Contingency Plan (MCP)			

1.0 Quality Control Requirements and Performance Standards for WSC-CAM-V C

1.1 Overview of WSC-CAM-V C

WSC-CAM-V C, *Quality Control Requirements and Performance Standards for the Analysis of Chlorinated Herbicides by Gas Chromatography (GC) in Support of Response Actions under the Massachusetts Contingency Plan (MCP)*, is a component of MassDEP's Compendium of Analytical Methods (CAM). Effective July 1, 2010, this revised CAM protocol, WSC-CAM-V C, replaces the original Chlorinated Herbicide GC CAM document, WSC-CAM-V C (effective date, May 28, 2004). Refer to WSC-CAM-I A for an overview of the CAM process. Please note that while this protocol must be followed on and after the effective date of July 1, 2010 for the purpose of "Presumptive Certainty," the revised protocol may be used optionally prior to its effective date upon its publication on April 15, 2010.

This document provides Quality Control (QC) requirements and performance standards to be used in conjunction with the required analytical method SW-846 8151A, analysis for chlorinated herbicides in aqueous and solid samples by GC preceded by conventional sample preparation methods via SW-846 Methods, as described in Section 1.3 of this protocol. The QC requirements and performance standards specified in this document in Table V C-1 together with the analytical procedures described in EPA SW-846 Method 8151A, *Chlorinated Herbicides by GC Using Methylation or Pentafluorobenzoylation Derivatization*, constitute the WSC-CAM-V C protocol. All protocols included in the CAM are considered "methods" published by the MassDEP pursuant to the provisions of 310 CMR 40.0017(2). Use of EPA SW-846 8151A is a "Presumptive Certainty" requirement of WSC-CAM-V C. Sample preservation, container and analytical holding time specifications for aqueous, soil, and sediment matrices for chlorinated herbicides analyzed in support of MCP decision-making are presented in Appendix V C-1 of this document and Appendix VII-A of WSC-CAM-VII A *Quality Assurance and Quality Control Guidelines for the Acquisition and Reporting of Analytical Data in Support of Response Actions Conducted Under the Massachusetts Contingency Plan (MCP)*. Data reporting requirements are also provided in WSC-CAM-VII A.

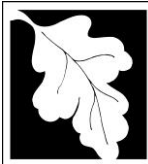
Overall usability of data produced using this CAM protocol should be evaluated for compliance with project-specific data quality objectives, regardless of "Presumptive Certainty" status. For more guidance on data usability, refer to MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*.

1.1.1 Reporting Limits for WSC-CAM-V C

The reporting limit (RL) for an individual compound using WSC-CAM-V C dependent on the concentration of the lowest non-zero standard in the initial calibration, analyzed under identical conditions as the sample, with adjustments made for the sample size, extraction concentration factor, percent solids, dilution factors, etc., as required. Except as provided in the table below, the CAM RLs for WSC-CAM-V C target analytes are:

- 0.5-2 µg/L for aqueous samples (surface water, groundwater and drinking water); and
- 5-80 µg/kg (wet weight) for soil/sediment samples (assuming 100% solids).

These values are readily achievable using electron capture detectors (ECDs). There may be exceptions to the above CAM RLs for some target analytes (that is, the CAM RL for some target analytes may not be readily

	Massachusetts Department of Environmental Protection Bureau of Waste Site Cleanup	WSC-CAM	Section: V C
		July 1, 2010	Revision No. 1
		Final	Page 5 of 29
Quality Control Requirements and Performance Standards for the <i>Analysis of Chlorinated Herbicides by Gas Chromatography (GC)</i> in Support of Response Actions under the Massachusetts Contingency Plan (MCP)			

achieved by a laboratory using WSC-CAM-V C). These CAM RL exceptions for the WSC-CAM-V C target analytes are presented in the table below for various matrices. For “Presumptive Certainty” purposes, if the CAM RLs are not achieved, respond “NO” to in Question G of the “MassDEP MCP Analytical Protocol Certification Form” and address the CAM RL exceedance in the laboratory narrative.

CAM RL Exceptions for WSC-CAM-V C Target Analytes		
Target Analyte	Groundwater/Surface Water ($\mu\text{g/L}$)	Soil/Sediment ¹ ($\mu\text{g/kg}$)
MCP	100	3,300
MCPA	100	3,300

¹ Assuming 100% solids

Reporting limits lower than the above-referenced CAM RLs for WSC-CAM-V C target analytes may be required to satisfy project requirements. The RL (based on the concentration of the lowest calibration standard) for each contaminant of concern must be less than or equal to the MCP standards or criteria that the contaminant concentrations are being compared to (e.g., Method 1 Standards, benchmark values, background, etc.). Meeting MCP standards or criteria may require method modifications, such as reducing the volume of the final extract, to improve sensitivity. All such modifications must be described in the laboratory narrative. Regardless of the modification that is used, RLs for the WSC-CAM-V C target analytes will be proportionately higher for samples that require dilution, when a reduced sample size is used, or for an increased final extract volume.

1.1.2 Initial Demonstration of Proficiency for WSC-CAM-V C

Each laboratory that uses the WSC-CAM-V C protocol is required to operate a formal quality assurance program. The minimum requirements of this program consist of an initial demonstration of laboratory proficiency, ongoing analysis of standards and blanks to confirm acceptable continuing performance, and the analysis of laboratory control samples (LCSs) and LCS duplicates to assess analytical accuracy and precision. Matrix spikes (MS), matrix spike duplicates (MSD) or matrix duplicates may also be used to evaluate accuracy and precision when such samples are analyzed either at the discretion of the laboratory or at the request of the data user.

Laboratories must document and have on file an Initial Demonstration of Proficiency for each combination of sample preparation and determinative method being used. These data must meet or exceed the performance standards as presented in Table V C-1 of this protocol and SW-846 Method 8000B. Procedural requirements for performing the Initial Demonstration of Proficiency can be found in SW-846 Method 8000B (Section 8.4) and SW-846 method 8151A (Section 8.3). The data associated with the Initial Demonstration of Proficiency must be kept on file at the laboratory and made available to potential data users on request. The data associated with the Initial Demonstration of Proficiency for WSC-CAM-V C must include the following information:



Quality Control Requirements and Performance Standards for the ***Analysis of Chlorinated Herbicides by Gas Chromatography (GC)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

QC Element	Performance Criteria
Initial Calibration	WSC-CAM-V C, Table V C-1
Continuing Calibration	WSC-CAM-V C, Table V C-1
Method Blanks	WSC-CAM-V C, Table V C-1
Average Recovery	SW-846 Method 8000B, Section 8.4
% Relative Standard Deviation	SW-846 Method 8000B, Section 8.4
Surrogate Recovery	WSC-CAM-V C, Table V C-1
Internal Standards	WSC-CAM-V C, Table V C-1

NOTE: Because of the number of QC elements associated with the Initial Demonstration of Proficiency, it should be expected that one or more analytes may not meet the performance standard for one or more QC elements. Under these circumstances, the analyst should attempt to locate and correct the problem and repeat the analysis for all non-conforming analytes. All non-conforming analytes along with the laboratory-specific acceptance criteria should be noted in the Initial Demonstration of Proficiency documentation.

It is essential that laboratory-specific performance criteria for LCS, LCS duplicate and surrogate recoveries also be calculated and documented as described in SW-846 Method 8000B, Section 8.7. Experience indicates that the criteria recommended in specific methods are frequently not met for some analytes and/or matrices; the in-house performance criteria will be a means of documenting these repeated exceedances. Laboratories are encouraged to actively monitor pertinent QC performance standards described in Table V C-1 to assess analytical trends (i.e., systematic bias, etc) and improve overall method performance by preempting potential non-conformances.

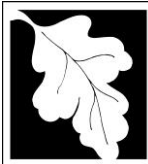
For the WSC-CAM-V C protocol, laboratory-specific control limits must meet or exceed (demonstrate less variability than) the performance standards for each QC element listed in Table V C-1. It should be noted that the performance standards listed in Table V C-1 are based on multiple-laboratory data, which are in most cases expected to demonstrate more variability than performance standards developed by a single laboratory.

This protocol is restricted to use by, or under the supervision of, analysts experienced in the use of GC instrumentation as a quantitative tool and skilled in the interpretation of chromatograms for chlorinated herbicides.

1.2 Summary of SW-846 Method 8151A

A measured volume or weight of sample (approximately 1 L for liquids, 30 to 50 grams [dry weight] for solids) is extracted using the appropriate extraction technique (see Section 1.3).

- Aqueous samples are extracted at a pH >12.0 with methylene chloride with manual shaking or mechanical techniques. The pH of the extracted sample is adjusted to <2.0 and extracted with

	Massachusetts Department of Environmental Protection Bureau of Waste Site Cleanup	WSC-CAM	Section: V C
		July 1, 2010	Revision No. 1
		Final	Page 7 of 29
Quality Control Requirements and Performance Standards for the <i>Analysis of Chlorinated Herbicides by Gas Chromatography (GC)</i> in Support of Response Actions under the Massachusetts Contingency Plan (MCP)			

diethyl ether in a separatory funnel. The extract is then concentrated and derivitized prior to analysis.

- Solid samples are extracted with a methylene chloride/acetone mixture (1:1 v/v) after acidification with concentrated hydrochloric acid (pH >2.0) or phosphate buffer (pH 2.5). Sample hydrolysis and/or cleanup are applied as necessary as described in Sections 7.2.3 and 7.2.4 of SW-846 method 8151A, respectively. The extract is then concentrated and derivitized prior to analysis.

The derivitized extract is analyzed by injecting a 1 to 2- μ L aliquot into a gas chromatograph with a narrow- or wide-bore fused silica capillary column equipped with an ECD. The GC oven is temperature-programmed to facilitate separation of the analytes of interest, which are then detected by an ECD that is interfaced directly to the GC.

Identification of target analytes is accomplished by comparing the sample retention time with the retention time of standards obtained under identical analytical conditions. Quantitation is accomplished by using the peak area and a calibration factor generated from a minimum five-point calibration curve.

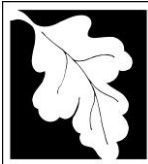
Identification of chlorinated herbicides on a single-column must be confirmed on a second column, or must be supported by at least one other independent qualitative technique. Although a dual-column option may satisfy this requirement, due caution should be exercised when highly contaminated samples are processed or during times of high sample throughput. Dual column confirmation is not required for samples with concentrations of all chlorinated herbicides below their respective RL.

1.3 Sample Extraction/Cleanup Methods for WSC-CAM-V C

Samples for analysis by SW-846 Method 8151A must be extracted using one of the following methods.

Matrix	Method
Aqueous	Refer to Section 7.3 of SW-846 Method 8151A
Soil/Sediment	Ultrasonic or Shaker Extraction, refer to Section 7.2 of SW-846 Method 8151A
Waste	SW-846 Method 3580, Waste Dilution, refer to Section 7.1 of SW-846 Method 8151A
Extract Concentration (Solid & Aqueous Samples)	Refer to Section 7.4 of SW-846 Method 8151A
Derivatization (Solid & Aqueous Samples)	Refer to Section 7.5 of SW-846 Method 8151A. Bubbler Method, Section 7.5.1.1 is recommended for samples with low concentrations and the Diazald Kit Method, Section 7.5.1.2 is recommended for solid samples and samples with high concentrations.

If additional cleanup of chlorinated herbicide extracts (only applicable to non-hydrolyzed species) is required, refer to Section 7.2.4 of SW-846 Method 8151A.


	Massachusetts Department of Environmental Protection Bureau of Waste Site Cleanup	WSC-CAM	Section: V C
		July 1, 2010	Revision No. 1
		Final	Page 8 of 29
Quality Control Requirements and Performance Standards for the <i>Analysis of Chlorinated Herbicides by Gas Chromatography (GC)</i> in Support of Response Actions under the Massachusetts Contingency Plan (MCP)			

1.4 Method Interferences

- Refer to SW-846 Methods 3500C (Section 4.0, in particular), 3600C, and 8000B for a detailed discussion of interferences. Interferences co-extracted from the samples will vary considerably from matrix to matrix. While general cleanup techniques are referenced or provided as part of this method, unique samples may require additional cleanup approaches to achieve desired degrees of discrimination and quantitation. Sources of interference in this method can be grouped into four broad categories.
 - Contaminated solvents, reagents, or sample processing hardware,
 - Contaminated GC carrier gas, parts, column surfaces, or detector surfaces,
 - Non-target compounds simultaneously extracted from the sample matrix which cause a detector response, and
 - Co-elution of target analytes.

An in depth discussion of the causes and corrective actions for all of these interferences is beyond the scope of this guidance document. A brief discussion of the more prevalent interferences is presented below.

- Refer to SW-846 Method 8151A for a detailed description of chemical contaminants, cross-contamination, and corrective actions which may be taken to eliminate contamination. If a method blank contains a contaminant, data for samples associated with that blank must **not** undergo "blank correction" (i.e., if an associated sample also contains the contaminant, subtraction of the blank amount from the sample amount is not permitted).
- Organic acids, especially chlorinated acids, cause the most direct interferences with the determination by methylation. Phenols, including chlorophenols, may also interfere with this procedure. The determination using pentafluorobenzoylation is more sensitive, and more prone to interferences from the presence of organic acids or phenols than by methylation. Herbicides, being strong organic acids, react readily with alkaline substances and may be lost during analysis. As a precaution, glassware should be acid-rinsed and then re-rinsed to constant pH with organic-free water. **All sodium sulfate used for drying must be acidified.** Other common interferences are presented in Section 3.0 of SW-846 Method 8151A.
- Cross-contamination may occur when any sample is analyzed immediately after a sample containing high concentrations of chlorinated herbicides. After the analysis of a sample containing high concentrations of chlorinated herbicides, one or more blanks should be analyzed to check for potential cross-contamination/carryover. Concentrations of chlorinated herbicides which exceed the upper limit of calibration should prompt the analyst to check for potential cross-contamination/carryover. In addition, samples containing large amounts of water-soluble materials, suspended solids, or high boiling point compounds may also present potential for cross-contamination/carryover. Laboratories should be aware that carryover from high boiling point compounds may not appear until a later sample analysis. To reduce carryover, the sample syringe must be rinsed with solvent between sample injections.

	Massachusetts Department of Environmental Protection Bureau of Waste Site Cleanup	WSC-CAM	Section: V C
		July 1, 2010	Revision No. 1
		Final	Page 9 of 29
Quality Control Requirements and Performance Standards for the <i>Analysis of Chlorinated Herbicides by Gas Chromatography (GC)</i> in Support of Response Actions under the Massachusetts Contingency Plan (MCP)			

1.5 Quality Control Requirements for WSC-CAM-V C

1.5.1 General QC Requirements

Refer to SW-846 Method 8000B for general QC procedures for all chromatographic methods, which includes SW-846 method 8151A. Instrument QC and method performance requirements for the GC/ECD system may be found in SW-846 method 8151A, Sections 9.0 and 13.0, respectively.

1.5.2 Specific QC Requirements and Performance Standards for WSC-CAM-V C

Specific QC requirements and performance standards for the WSC-CAM-V C protocol are presented in Table V C-1. Refer to WSC-CAM-VII A for field QC requirements. Strict compliance with the QC requirements and performance standards, as well as satisfying the CAM's other analytical and reporting requirements will provide a data user with "Presumptive Certainty" in support of Response Actions under the MCP. The concept of "Presumptive Certainty" is explained in detail in Section 2.0 of WSC-CAM-VII A.

While optional, parties electing to utilize these protocols will be assured of "Presumptive Certainty" of data acceptance by agency reviewers. In order to achieve "Presumptive Certainty" for analytical data, parties must:

- Use the analytical method specified for the selected CAM protocol;
- Incorporate **all** required analytical QC elements specified for the selected CAM protocol;
- Implement, as necessary, required corrective actions and analytical response actions for **all** non-conforming analytical performance standards;
- Evaluate and narrate, as necessary, **all** identified CAM protocol non-compliances; and
- Comply with **all** the reporting requirements specified in WSC-CAM-VII A, including retention of reported and unreported analytical data and information for a period of ten (10) years.

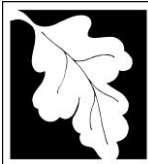
In achieving "Presumptive Certainty" status, parties will be assured that analytical data sets:

- ✓ Satisfy the broad QA/QC requirements of 310 CMR 40.0017 and 40.0191 regarding the scientific defensibility, precision and accuracy, and reporting of analytical data; and
- ✓ May be used in a data usability and representativeness assessment, as required in 310 CMR 40.1056(2)(k) for Response Action Outcome (RAO) submittals, consistent with the guidance described in MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*.

1.6 Special Analytical Considerations for WSC-CAM-V C

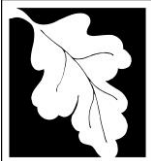
The following bullets highlight potential issues that may be encountered with the analysis of chlorinated herbicides using this protocol.

- Sample extracts should be scrupulously dried prior to methylation to avoid poor recoveries.
- Alkaline hydrolysis and subsequent extraction of the basic solution removes many chlorinated hydrocarbons and phthalate esters that might otherwise interfere with ECD analysis. However,

	Massachusetts Department of Environmental Protection Bureau of Waste Site Cleanup	WSC-CAM	Section: V C
		July 1, 2010	Revision No. 1
		Final	Page 10 of 29
Quality Control Requirements and Performance Standards for the <i>Analysis of Chlorinated Herbicides by Gas Chromatography (GC)</i> in Support of Response Actions under the Massachusetts Contingency Plan (MCP)			

hydrolysis may result in the loss of Dinoseb and the formation of interfering aldol condensation products, if any residual acetone remains from the extraction of solids.

- The diazomethane solution used must be freshly prepared. Only experienced analysts should be allowed to work with diazomethane due to the potential hazards associated with its use (i.e., explosive, carcinogenic).
- Esterification duration is critical to the herbicide recoveries. Methylated extracts are subject to trans-esterification and other unwanted side reactions.
- If calibration standards are prepared using methyl esters, the calculation of concentration must include a correction for the molecular weight of the methyl ester versus the acid herbicide.
- Sample extracts must be analyzed immediately after the methylation procedure has been performed in order to minimize the trans-esterification and other potential reactions that may occur. This is critical in the evaluation of whether the holding time criteria were achieved.
- A linear or non-linear calibration model must not be used to compensate for detector saturation or to avoid proper instrument maintenance. As such, linear or non-linear regression must not be employed for initial calibration calculations that typically meet percent relative standard deviation (%RSD) requirements specified in Table V C-1.



Massachusetts Department of Environmental
Protection Bureau of Waste Site Cleanup

WSC-CAM

Section: V C

July 1, 2010

Revision No. 1

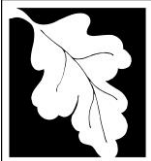
Final

Page 11 of 29

Quality Control Requirements and Performance Standards for the ***Analysis of Chlorinated Herbicides by Gas Chromatography (GC)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

Table V C-1: Specific QC Requirements and Performance Standards for Chlorinated Herbicides (SW-846 8151A) Using WSC-CAM-V C

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350 ¹	Required Corrective Action	Required Analytical Response Action
Initial Demonstration of Proficiency	Laboratory Analytical Accuracy & Precision	(1) Must be performed prior to using method on samples. (2) Must be performed for each matrix. (3) Must contain all target analytes. (4) Must follow procedure in Section 8.4 of SW-846 8000B.	No	NA	Refer to Section 8.4 of SW-846 8000B and Section 1.1.2 of this protocol	NA
Retention Time Windows	Laboratory Analytical Accuracy	(1) Prior to initial calibration and when a new GC column is installed. (2) Calculated according to the method (Section 7.6 of SW-846 8000B)	No	NA	NA	NA
Initial Calibration	Laboratory Analytical Accuracy	(1) Must be analyzed at least once prior to analyzing samples, when initial calibration verification or continuing calibration does not meet the performance standards, and when major instrument maintenance is performed. (2) Minimum of 5 standards (or 6 if non-linear regression used). (3) Low standard must be \leq RL. (4) $\%RSD \leq 20$, $r \geq 0.99$ (linear regression), or $r^2 \geq 0.99$ (non-linear regression) for each target herbicide. (5) If $\%RSD > 20$, linear or non-linear regression must be used. (6) Must contain all target herbicides. (7) Calibration must be performed under the same conditions as the samples. (8) If linear or non-linear regression used, verify the RL by recalculating concentrations in lowest calibration standard using the final calibration curve; recoveries must be 70-130%.	No	NA	(1) Recalibrate as required by method. (2) If recalculated concentrations from the lowest calibration standard are outside of 70-130% recovery range, either: * The RL limit must be reported as an estimated value ² , or * The RL must be raised to the concentration of the next highest calibration standard that exhibits acceptable recoveries when recalculated using the final calibration curve.	Sample analysis cannot proceed without a valid initial calibration. Report non-conforming compounds ($\%RSD > 20$, $r < 0.99$, or $r^2 < 0.99$) in laboratory narrative. If non-linear regression (i.e., quadratic equation) is used for calibration, this must be noted in the laboratory narrative along with the compounds affected.



Massachusetts Department of Environmental
Protection Bureau of Waste Site Cleanup

WSC-CAM

Section: V C

July 1, 2010

Revision No. 1

Final

Page 12 of 29

Quality Control Requirements and Performance Standards for the ***Analysis of Chlorinated Herbicides by Gas Chromatography (GC)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

Table V C-1: Specific QC Requirements and Performance Standards for Chlorinated Herbicides (SW-846 8151A) Using WSC-CAM-V C

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350 ¹	Required Corrective Action	Required Analytical Response Action
Initial Calibration Verification	Laboratory Analytical Accuracy	(1) Immediately after each initial calibration. (2) Concentration level near midpoint of curve. (3) Prepared using standard source different than used for initial calibration. (4) Must contain all target herbicides. (5) Percent recoveries must be between 80-120% for each target analyte.	No	NA	Locate source of problem; recalibrate if >10% of all analytes are outside of criteria.	If recovery is outside of 80-120% for any analyte, report non-conforming compounds in laboratory narrative.
Continuing Calibration	Laboratory Analytical Accuracy	(1) Prior to samples, every 12 hours or every 10 samples, whichever is more frequent, and at the end of the analytical sequence. (NOTE: if internal standard calibration used, the continuing calibration at the end of the analytical sequence is not required). (2) Concentration level near midpoint of curve. (3) Must contain all target herbicides. (4) %D must be ≤ 15 for each target analyte. (5) Verify that all analytes fall within retention time windows. (6) Area count of internal standard in continuing calibration must be within $\pm 50\%$ of the average area count in the associated initial calibration.	No	NA	(1) Perform instrument maintenance, reanalyze continuing calibration and/or recalibrate as required by method. (2) Reanalyze "associated samples" if beginning or ending continuing calibration exhibited low response. (3) Reanalyze "associated samples" if beginning or ending continuing calibration exhibited high response and associated herbicides were detected in the "associated samples." NOTE: "Associated samples" refers to all samples analyzed since the last acceptable continuing calibration.	Report non-conforming compounds (%D >15) and associated samples in laboratory narrative.



Massachusetts Department of Environmental
Protection Bureau of Waste Site Cleanup

WSC-CAM

Section: V C

July 1, 2010

Revision No. 1

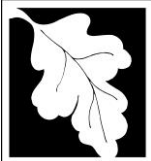
Final

Page 13 of 29

Quality Control Requirements and Performance Standards for the ***Analysis of Chlorinated Herbicides by Gas Chromatography (GC)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

Table V C-1: Specific QC Requirements and Performance Standards for Chlorinated Herbicides (SW-846 8151A) Using WSC-CAM-V C

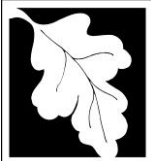
Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350 ¹	Required Corrective Action	Required Analytical Response Action
Method Blank	Laboratory Method Sensitivity (contamination evaluation)	(1) Extracted with every batch or every 20 samples, whichever is more frequent. (2) Matrix-specific (e.g., water, soil). (3) Target analytes must be <RL.	Yes	NA	(1) If concentration of contaminant in sample is <10x concentration in blank, locate source of contamination; correct problem; re-extract and re-analyze method blank and associated samples. (2) No corrective action required if concentration of contaminant in sample is >10x concentration in blank or if contaminant not detected in sample.	(1) If sample re-extraction is not possible, report non-conformance in laboratory narrative. (2) If contamination of method blanks is suspected or present, the laboratory, using a "B" or some other convention, should qualify the sample results. Blank contamination should also be documented in the laboratory narrative. (3) If re-extraction is performed within holding time and yields acceptable method blank results, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time, the laboratory must report results of both the initial extraction and re-extraction.



Quality Control Requirements and Performance Standards for the ***Analysis of Chlorinated Herbicides by Gas Chromatography (GC)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

Table V C-1: Specific QC Requirements and Performance Standards for Chlorinated Herbicides (SW-846 8151A) Using WSC-CAM-V C

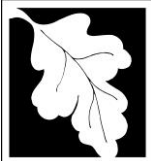
Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350 ¹	Required Corrective Action	Required Analytical Response Action
Laboratory Control Sample (LCS)	Laboratory Analytical Accuracy	(1) Extracted with every batch or every 20 samples, whichever is more frequent. (2) Concentration level near midpoint of curve. (3) Must contain all target herbicides. (4) Matrix-specific (e.g., soil, water). (5) Percent recoveries must be between 40-140% for all target analytes with the exception of Dinoseb. Laboratories must develop in-house recovery control limits for Dinoseb. (6) Must be prepared in a water-miscible solvent (e.g., acetone, methanol).	Yes	Recovery <10%; affects nondetect results for affected analyte in all samples extracted with this LCS.	(1) Locate source of problem; re-extract and re-analyze LCS and associated samples if >10% of all analytes are outside of criteria. (2) If ≤10% of compounds are outside of the acceptance criteria, re-extraction is not required as long as recoveries are >10%. (3) If >10% of compounds are above the acceptance criteria (>140%), reextraction is not required if affected compounds were not detected in associated samples.	(1) If sample re-extraction is not possible, report non-conformance in laboratory narrative. (2) If recovery is outside of 40-140% for any analyte, report non-conforming compounds in laboratory narrative. (3) If re-extraction is performed within holding time and yields acceptable LCS results, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time, the laboratory must report results of both the initial extraction and re-extraction.
LCS Duplicate	Laboratory Analytical Accuracy & Precision	(1) Extracted with every batch or every 20 samples, whichever is more frequent. (2) Concentration level near midpoint of curve. (3) Must contain all target herbicides. (4) Matrix-specific (e.g., soil, water). (5) Percent recoveries must be between 40-140% for all target analytes with the exception of Dinoseb. Laboratories must develop in-house recovery control limits for Dinoseb.	Yes	Recovery <10%; affects nondetect results for affected analyte in all samples extracted with this LCS.	(1) Locate source of problem; re-extract and re-analyze LCS and associated samples if >10% of all analytes are outside of recovery acceptance criteria. (2) If ≤10% of compounds are outside of the recovery acceptance criteria, re-extraction is not required as long as	(1) If sample re-extraction is not possible, report non-conformance in laboratory narrative. (2) If recovery is outside of 40-140% for any analyte or if RPD is outside of criteria, report non-conforming compounds in laboratory narrative.



Quality Control Requirements and Performance Standards for the ***Analysis of Chlorinated Herbicides by Gas Chromatography (GC)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

Table V C-1: Specific QC Requirements and Performance Standards for Chlorinated Herbicides (SW-846 8151A) Using WSC-CAM-V C

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350 ¹	Required Corrective Action	Required Analytical Response Action
		(6) RPDs must be ≤ 20 for waters and ≤ 30 for solids. (7) Must be prepared in a water-miscible solvent (e.g., acetone, methanol).			recoveries are $>10\%$. (3) If $>10\%$ of compounds are above the recovery acceptance criteria ($>140\%$), reextraction is not required if affected compounds were not detected in associated samples.	(3) If re-extraction is performed within holding time and yields acceptable LCS results, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of holding time, the laboratory must report results of both the initial extraction and re-extraction.
MS/MSD	Method Accuracy & Precision in Sample Matrix	(1) Every 20 samples (at discretion of laboratory or at request of data user). (2) Matrix-specific. (3) Concentration level near midpoint of curve. (4) Must contain all target herbicides. (5) Percent recoveries between 30 – 150%. (6) RPDs ≤ 20 for waters and ≤ 30 for solids. (7) Must be prepared in a water-miscible solvent (e.g., acetone, methanol).	Yes ONLY when requested by the data user	Recovery $<10\%$; affects nondetect result for affected analyte in unspiked sample only.	Check LCS; if recoveries are acceptable in LCS, narrate non-conformance.	Note exceedances in laboratory narrative.
Surrogates	Method Accuracy in Sample Matrix	(1) Minimum of 1 surrogate. Recommended surrogate: DCAA (2) Percent recoveries must be between 30-150% on both columns.	Yes (report surrogate recoveries from both columns)	Recovery $<10\%$; affects all nondetect results in affected sample.	If the surrogate is outside of limits on both columns: (1) Re-extract the sample if surrogate recoveries are low and there is no chromatographic interference. (2) Re-extract the sample if surrogate recoveries are high and herbicides were detected in the sample.	(1) Report recoveries outside of 30-150% in laboratory narrative. (2) If re-extraction yields similar surrogate non-conformances, the laboratory must report results of both the initial extraction and re-extraction. (3) If re-extraction is



Massachusetts Department of Environmental
Protection Bureau of Waste Site Cleanup

WSC-CAM

Section: V C

July 1, 2010

Revision No. 1

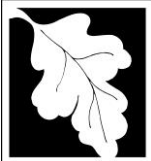
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Page 16 of 29

Quality Control Requirements and Performance Standards for the ***Analysis of Chlorinated Herbicides by Gas Chromatography (GC)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

Table V C-1: Specific QC Requirements and Performance Standards for Chlorinated Herbicides (SW-846 8151A) Using WSC-CAM-V C

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350 ¹	Required Corrective Action	Required Analytical Response Action
					NOTES: (a) If surrogate recoveries are high and target analytes are not detected in sample, re-extraction is not required. (b) If chromatographic interference is present and surrogate recovery would cause rejection of data (i.e., <10%), reanalyze sample on dilution. (c) If a surrogate is diluted to a concentration below that of the lowest calibration standard, reextraction and/or reanalysis is not required.	performed within holding time and yields acceptable surrogate recoveries, the laboratory may report results of the re-extraction only. (4) If re-extraction is performed outside of the holding time and yields acceptable surrogate recoveries, the laboratory must report results of both the initial extraction and re-extraction. (5) If sample is not re-extracted due to chromatographic interference, the laboratory must provide the chromatogram in the data report.
Internal Standards (optional)	Laboratory Analytical Accuracy and Method Accuracy in Sample Matrix	(1) Minimum of 1. Recommended internal standard: DBOFB (2) Area counts in samples must be between 50 – 200% of the area counts in the associated continuing calibration standard. (3) Retention times of internal standards must be within ± 30 seconds of retention times in associated continuing calibration standard.	No	Recovery <20%; affects all nondetect results quantitated using affected internal standard in associated sample.	If internal standard is outside of limits, reanalyze sample unless chromatographic interference present. NOTE: If chromatographic interference is present and internal standard area would cause rejection of data (i.e., <20%), reanalyze sample on dilution.	(1) Report nonconformances in laboratory narrative. Include actual recovery of internal standard and provide summary of analytes quantitated using the internal standard. (2) If reanalysis yields similar internal standard non-conformances, the laboratory must report results of both analyses. (3) If reanalysis is



Massachusetts Department of Environmental
Protection Bureau of Waste Site Cleanup

WSC-CAM

Section: V C

July 1, 2010

Revision No. 1

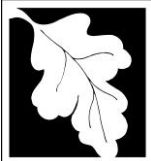
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Page 17 of 29

Quality Control Requirements and Performance Standards for the ***Analysis of Chlorinated Herbicides by Gas Chromatography (GC)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

Table V C-1: Specific QC Requirements and Performance Standards for Chlorinated Herbicides (SW-846 8151A) Using WSC-CAM-V C

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350 ¹	Required Corrective Action	Required Analytical Response Action
						performed within holding time and yields acceptable internal standard recoveries, the laboratory may report results of the reanalysis only. (4) If reanalysis is performed outside of the holding time and yields acceptable internal standard recoveries, the laboratory must report results of both analyses. (5) If sample is not reanalyzed due to chromatographic interference, the laboratory must provide the chromatogram in the data report.
Identification and Quantitation	NA	(1) Peak area is the expected default to be used for quantitation of herbicides under most circumstances. Regardless if peak area or peak height is used, the same method used for quantitation of samples must also be used for calibration standards. (2) The laboratory must use the average calibration factor, response factor, or linear or non-linear regression curve generated from the associated initial calibration for quantitation of each target herbicide. (3) Secondary column analysis: Laboratory must utilize a second dissimilar column to confirm positive results. The laboratory must report the higher of	NA	If RPD >100 for target herbicides, reject positive result for affected herbicide.	If the RPD between the dual column results is >100, reanalyze the sample on dilution. Both analyses must be reported. Alternatively, additional sample cleanup techniques may be warranted.	If the RPD between the dual column results exceeds 40, the laboratory must qualify the sample results and/or note the exceedance in the laboratory narrative. If the RPD exceedance is due to interference, the lower of the dual column values can be reported; this must be noted in the laboratory narrative.



Massachusetts Department of Environmental
Protection Bureau of Waste Site Cleanup

WSC-CAM

Section: V C

July 1, 2010

Revision No. 1

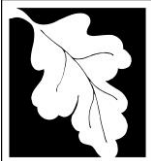
Final

Page 18 of 29

Quality Control Requirements and Performance Standards for the ***Analysis of Chlorinated Herbicides by Gas Chromatography (GC)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

Table V C-1: Specific QC Requirements and Performance Standards for Chlorinated Herbicides (SW-846 8151A) Using WSC-CAM-V C

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350 ¹	Required Corrective Action	Required Analytical Response Action
		the two results. All required QC parameters (e.g., calibrations, LCSs, etc.) must be met on the secondary column as well. (4) Results must be reported with 2 or more "significant figures" if \geq RL. If reporting values below the RL, report with 1 or more "significant figures". ³				
General Reporting Issues	NA	(1) The laboratory must only report values \geq the sample-specific reporting limit. (2) Dilutions: If diluted and undiluted analyses are performed, the laboratory should report results for the lowest dilution within the valid calibration range for <u>each</u> analyte. The associated QC (e.g., method blanks, surrogates, etc.) for each analysis must be reported. (3) Results for soils/sediments must be reported on a dry-weight basis for comparison to MCP regulatory standards. (4) Refer to Appendix V C-1 for chain-of-custody requirements regarding preservation, cooler temperature, and holding times.	NA	NA	NA	(1) Complete analytical documentation for diluted and undiluted analyses must be made available for review during an audit. (2) The performance of dilutions must be documented in the laboratory narrative or on the report form. Unless due to elevated concentrations of target compounds, reasons for dilutions must be explained in the laboratory narrative. (3) If samples are not preserved properly or are not received with an acceptable cooler temperature, note the non-conformances in the laboratory narrative. (4) If samples are extracted and/or analyzed outside of the holding time, note the non-conformances in the laboratory narrative.



Massachusetts Department of Environmental
Protection Bureau of Waste Site Cleanup

WSC-CAM

Section: V C

July 1, 2010

Revision No. 1

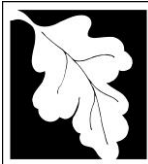
Final

Page 19 of 29

Quality Control Requirements and Performance Standards for the ***Analysis of Chlorinated Herbicides by Gas Chromatography (GC)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

Table V C-1: Specific QC Requirements and Performance Standards for Chlorinated Herbicides (SW-846 8151A) Using WSC-CAM-V C

Required QC Parameter	Data Quality Objective	Required Performance Standard	Required Deliverable?	Rejection Criteria per WSC-07-350 ¹	Required Corrective Action	Required Analytical Response Action
<p>¹As per Appendix IV of MassDEP Policy #WSC-07-350, <i>MCP Representativeness Evaluations and Data Usability Assessments</i>, September 2007, if these results are observed, data users should consider nondetect results as unusable and positive results as estimated with a significant low bias.</p> <p>²If the RL is estimated due to unacceptable recovery of the lowest standard, the CAM RL has not been achieved; Question G of the “MassDEP MCP Analytical Protocol Certification Form” must be answered “NO” and this must be addressed in the laboratory narrative.</p> <p>³Reporting protocol for “significant figures” is a policy decision included for standardization and consistency for reporting of results and is not a definition of “significant” in the scientific or mathematical sense.</p>						

	Massachusetts Department of Environmental Protection Bureau of Waste Site Cleanup	WSC-CAM	Section: V C
		July 1, 2010	Revision No. 1
		Final	Page 20 of 29
Quality Control Requirements and Performance Standards for the <i>Analysis of Chlorinated Herbicides by Gas Chromatography (GC)</i> in Support of Response Actions under the Massachusetts Contingency Plan (MCP)			

1.7 Analyte List for WSC-CAM-V C

The MCP analyte list for WSC-CAM-V C is presented in Table V C-2. The list is comprised of potential contaminants that are readily-analyzable by WSC-CAM-V C.

It is the responsibility of the data user, in concert with the laboratory, to establish the range and required RL for the target analytes. Sources of various MassDEP standards and criteria are as follows:

- Reportable Quantities (RQs) and Concentrations (RCs) as described in 310 CMR 40.1600, The Massachusetts Oil and Hazardous Materials List (MOHML), in Subpart P of the MCP may be found at the following URL: <https://www.mass.gov/site-cleanup-regulations-policies-forms-more>.
- An online searchable Oil & Hazardous Materials List of RQs and RCs values may be found at the following URL: <https://www.mass.gov/service-details/oil-hazardous-material-list>.
- An updated list of MCP Method 1 Standards may be found at the following URL: <https://www.mass.gov/site-cleanup-regulations-policies-forms-more>.

None of the analytes listed in Table V C-2 have a promulgated MCP Method 1 groundwater/soil standard. However, each of the analytes listed in Table V C-2 has an MCP Reportable Concentration.

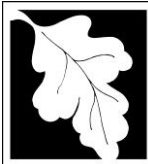
1.7.1 Analyte List Reporting Requirements for WSC-CAM-V C

While it is not necessary to request and report all the WSC-CAM-V C analytes listed in Table V C-2 to obtain "Presumptive Certainty" status, it is necessary to document use and reporting of a reduced analyte list, for site characterization and data representativeness considerations. MassDEP strongly recommends use of the full analyte list during the initial stages of site investigations, and/or at sites with an unknown or complicated history of uses of oil or hazardous materials. These assessment activities may include but are not limited to:

- ✓ Immediate Response Actions (IRAs) performed in accordance with 310 CMR 40.0410;
- ✓ Initial Site Investigation Activities performed in accordance with 310 CMR 40.0405(1);
- ✓ Phase I Initial Site Investigation Activities performed in accordance with 310 CMR 40.0480 through 40.0483; and
- ✓ Phase II Comprehensive Site Investigation Activities performed in accordance with 310 CMR 40.0830

In a limited number of cases, the use of the full analyte list for a chosen analytical method may not be necessary, with respect to data representativeness concerns, including:

- ✓ Sites where substantial site/use history information is available to rule-out all but a limited number of contaminants of concern, and where use of the full analyte list would significantly increase investigative costs; or
- ✓ Well-characterized sites where initial full-analyte list testing efforts have sufficiently narrowed the list of contaminants of concern.

	Massachusetts Department of Environmental Protection Bureau of Waste Site Cleanup	WSC-CAM	Section: V C
		July 1, 2010	Revision No. 1
		Final	Page 21 of 29
Quality Control Requirements and Performance Standards for the <i>Analysis of Chlorinated Herbicides by Gas Chromatography (GC)</i> in Support of Response Actions under the Massachusetts Contingency Plan (MCP)			

Note: a data user who avoids the detection and quantitation of a contaminant that is present or likely present at a site above background levels by limiting an analyte list could be found in criminal violation of MGL c. 21E or any regulations or orders adopted or issued thereunder.

In cases where a reduced list of analytes is requested, laboratories must still employ the specified QC requirements and performance standards in WSC-CAM-V C to obtain "Presumptive Certainty" status.



Massachusetts Department of Environmental
Protection Bureau of Waste Site Cleanup

WSC-CAM

Section: V C

July 1, 2010

Revision No. 1

Final

Page 22 of 29


Quality Control Requirements and Performance Standards for the ***Analysis of Chlorinated Herbicides by Gas Chromatography (GC)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

Table V C-2: Analyte List for WSC-CAM-V C (SW-846 8151A)

Analyte	CASN
2,5-Dichloro-6-methoxybenzoic acid (Dicamba)	1918-00-9
2,4-Dichlorophenoxy acetic acid (2,4-D)	94-75-7
2,4-Dichlorophenoxy butyric acid (2,4-DB)	94-82-6
2-(2,4-Dichlorophenoxy) propionic acid (Dichloroprop)	120-36-5
2,2-Dichloro propionic acid (Dalapon)	75-99-0
2,4-Dinitro-6-sec-butylphenol (Dinoseb)	88-85-7
2-Methyl-4-chlorophenoxy acetic acid (MCPA)	94-74-6
2-(2-Methyl-4-chlorophenoxy) propionic acid (MCPP)	93-65-2
2,4,5- Trichlorophenoxy acetic acid (2,4,5-T)	93-76-5
2,4,5-Trichlorophenoxy propionic acid (Silvex)	93-72-1

CASN – Chemical Abstracts Service Numbers

NOTE: Other chlorinated herbicides may also be analyzed using the WSC-CAM-V C Protocol but are not considered part of the CAM target analyte list.

	Massachusetts Department of Environmental Protection Bureau of Waste Site Cleanup	WSC-CAM	Section: V C
		July 1, 2010	Revision No. 1
		Final	Page 23 of 29
Quality Control Requirements and Performance Standards for the <i>Analysis of Chlorinated Herbicides by Gas Chromatography (GC)</i> in Support of Response Actions under the Massachusetts Contingency Plan (MCP)			

2.0 Data Usability Assessment

Specific guidance applicable to all Class A, B or C RAO Statements, including partial RAOs, for preparation of Representativeness Evaluations and Data Usability Assessments pursuant to 310 CMR 40.1056(2)(k) of the MCP is provided in *MCP Representativeness Evaluations and Data Usability Assessments* (Policy #WSC-07-350). This document provides general information regarding the purpose and content of these required evaluations as a component of and in support of an RAO submittal. The most current version of this document may be found at the following URL: <https://www.mass.gov/site-cleanup-regulations-policies-forms-more>.

Overall usability of data produced using this CAM protocol should be evaluated for compliance with project-specific data objectives using MassDEP Policy #WSC-07-350, regardless of “Presumptive Certainty” status.

3.0 Reporting Requirements for WSC-CAM-V C

3.1 General Reporting Requirements for WSC-CAM-V C

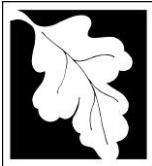
General environmental laboratory reporting requirements for analytical data used in support of assessment and evaluation decisions at MCP disposal sites are presented in WSC-CAM-VII A, Section 2.4. This guidance document provides limited recommendations for field QC, as well as the required content of the laboratory report, which includes:

- Laboratory identification information,
- Analytical results and supporting information,
- Sample- and batch-specific QC information,
- Laboratory Report Certification Statement,
- Copy of the Analytical Protocol Certification Form,
- Laboratory narrative contents, and
- Chain-of-custody form requirements.

3.2 Specific Reporting Requirements for WSC-CAM-V C

Specific QC requirements and performance standards for WSC-CAM-V C are presented in Table V C-1. Specific reporting requirements for WSC-CAM-V C are summarized below in Table V C-3 as “Required Analytical Deliverables (**YES**)”. These routine reporting requirements must always be included as part of the laboratory deliverable for this method. It should be noted that although certain items are not specified as “Required Analytical Deliverables (**NO**)”, these data must be available for review during an audit and may also be requested on a client-specific basis.

Soil and sediment results must be reported on a dry-weight basis. Refer to ASTM Method D2216, Determination of Moisture Content of Soils and Sediments, for more detailed analytical and equipment specifications.



Quality Control Requirements and Performance Standards for the ***Analysis of Chlorinated Herbicides by Gas Chromatography (GC)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

Table V C-3 Routine Reporting Requirements for WSC-CAM-V C (SW-846 8151A)

Parameter	Required Analytical Deliverable
Retention Time Windows	NO
Initial Calibration	NO
Initial Calibration Verification	NO
Continuing Calibration (CCAL)	NO
Method Blank	YES
Laboratory Control Samples (LCSs)	YES
LCS Duplicates	YES
Matrix Spike (MS)	YES (if requested by data user)
Matrix Spike Duplicate (MSD)	YES (if requested by data user)
Matrix Duplicate (MD)	YES (if requested by data user)
Surrogates	YES
Internal Standards	NO
Identification and Quantitation	NO
General Reporting Issues	YES

3.2.2 Sample Dilution


Under circumstances that sample dilution is required because either the concentration of one or more of the target analytes exceed the concentration of their respective highest calibration standard or any non-target peak exceeds the dynamic range of the detector (i.e., "off scale"), the RL for each chlorinated herbicide must be adjusted (increased) in direct proportion to the Dilution Factor (DF).

The revised RL for the diluted sample, RL_d :

$$RL_d = DF \times \text{Lowest Calibration Standard for Target Analyte}$$

It should be understood that samples with elevated RLs as a result of a dilution may not be able to satisfy MCP standards/criteria in some cases if the RL_d is greater than the applicable MCP standard or criterion to which the concentration is being compared. Such increases in RLs are the unavoidable but acceptable consequence of sample dilution that enable quantification of target analytes which exceed the calibration range. All dilutions must be fully documented in the laboratory narrative.

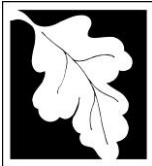
NOTE: Over dilution is an unacceptable laboratory practice. The post-dilution concentration of the target analyte with the highest concentration must be at least 60 to 80% of its associated highest calibration standard. This will avoid unnecessarily high RLs for other target analytes which did not require dilution.

	Massachusetts Department of Environmental Protection Bureau of Waste Site Cleanup	WSC-CAM	Section: V C
		July 1, 2010	Revision No. 1
		Final	Page 25 of 29
Quality Control Requirements and Performance Standards for the <i>Analysis of Chlorinated Herbicides by Gas Chromatography (GC)</i> in Support of Response Actions under the Massachusetts Contingency Plan (MCP)			

Appendix V C-1

Sample Collection, Preservation, and Handling Procedures for Chlorinated Herbicide Analyses

Sample preservation, container and analytical holding time specifications for aqueous, soil, and sediment matrices for chlorinated herbicides analyzed in support of MCP decision-making are summarized below and presented in Appendix VII A-1 of WSC-CAM-VII A, *Quality Assurance and Quality Control Guidelines for the Acquisition and Reporting of Analytical Data Conducted in Support of Response Actions Conducted Under the Massachusetts Contingency Plan (MCP)*.



Quality Control Requirements and Performance Standards for the ***Analysis of Chlorinated Herbicides by Gas Chromatography (GC)*** in Support of Response Actions under the Massachusetts Contingency Plan (MCP)

Matrix	Container ¹	Preservation ⁷	Holding Time ^{3,6}
Aqueous Samples, with no Residual Chlorine	(2) 1-L amber glass bottles w/ Teflon-lined screw caps	Cool to $\leq 6^{\circ}\text{C}$ but not frozen	7 days to extraction; 28 days to methylation; analysis immediately after methylation ⁵
Aqueous Samples, with Residual Chlorine ⁴	(2) 1-L amber glass bottles w/ Teflon-lined screw caps	Add 1-mL 10% sodium thiosulfate solution per container (or 0.008%) ⁴ . Addition of thiosulfate solution to sample container may be performed in the laboratory prior to field use. Cool to $\leq 6^{\circ}\text{C}$ but not frozen.	7 days to extraction; 28 days to methylation; analysis immediately after methylation ⁵
Soil/Sediment Samples	(1) 8-oz. amber glass jar w/ a Teflon-lined screw cap ²	Cool to $\leq 6^{\circ}\text{C}$ ²	14 days to extraction; 28 days to methylation; analysis immediately after methylation ^{2,5}
Waste Samples	(1) 500-mL amber wide mouth jar with a Teflon-lined screw cap.	No special preservation required	14 days to extraction; 28 days to methylation; analysis immediately after methylation ⁵

¹The number of sampling containers specified is not a requirement. For specific analyses, the collection of multiple sample containers is encouraged to avoid resampling if sample is consumed or compromised during shipping and/or analysis.

²Alternatively, soil/sediment samples for chlorinated herbicide analyses may be held for up to one (1) year if frozen within 24 hours of collection at $<-10^{\circ}\text{C}$. Sampling container should only be filled to 2/3 of capacity to avoid breakage caused by expansion during freezing. Preparation or extraction must be commenced within 24 hours of thawing. Temperature must never be allowed to go below -20°C to avoid damage to seals, etc.


³Holding time begins from time of sample collection or date thawed (see note #2 above).

⁴Presence of chlorine residual is usually associated with drinking water samples. Confirm dechlorination. If residual chlorine $>5\text{ mg/L}$, additional dechlorination agent may be required.

⁵Herbicide sample extracts must be stored at 4°C , protected from light, and stored in sealed vials (e.g., screw-cap or crimp-capped vials) with un-pierced PTFE-lined septa.

⁶As per Appendix IV of MassDEP Policy #WSC-07-350, *MCP Representativeness Evaluations and Data Usability Assessments*, September 2007, if the holding time is exceeded by $>2\times$ the allowable holding time, data users should consider nondetect results as unusable and positive results as estimated with a significantly low bias.

⁷If samples were received by the laboratory on the same day of collection and were stored and transported to the laboratory on ice, cooler temperatures above 6°C are acceptable.

	Massachusetts Department of Environmental Protection Bureau of Waste Site Cleanup	WSC-CAM	Section: V C
		July 1, 2010	Revision No. 1
		Final	Page 27 of 29
Quality Control Requirements and Performance Standards for the <i>Analysis of Chlorinated Herbicides by Gas Chromatography (GC)</i> in Support of Response Actions under the Massachusetts Contingency Plan (MCP)			

Appendix V C-2

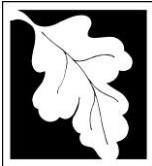
Data Deliverable Requirements for Data Audits



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If requested by MassDEP, submission of the information listed below may be required to perform a data audit to verify compliance with the analytical methods and to evaluate accuracy and reliability of the reported results. These deliverables represent a “full data package” including all sample documentation from receipt through preparation, analysis, and data reporting. The laboratory must ensure that these deliverables are available, in the event a data audit is performed. The laboratory is required to retain these deliverables for a period of 10 years from the date generated.

DELIVERABLE REQUIREMENTS FOR DATA AUDITS WSC-CAM-V C (Chlorinated Herbicides by GC/ECD)	
Laboratory Narrative	Must comply with the required laboratory narrative contents as described in WSC-CAM-VII A
Sample Handling Information	Chains-of-custody (external and internal), sample receipt logs (cooler temperatures and sample pH), correspondences
Miscellaneous Logs	Dry weight logs Injection logs Soil/sediment sample weight logs Freezer logs Sample preparation/cleanup logs ¹
Initial Calibration Data (both columns)	Summary of calibration factors for all standards in initial calibration; average calibration factors, %RSDs, correlation coefficients, and coefficients of determination for all target compounds Chromatograms for all standards used in initial calibration Quantitation reports for all standards used in initial calibration Concentrations of standards used must be clearly presented
Initial Calibration Verification Data (both columns)	Summary of percent recoveries for all target compounds Chromatograms for all ICVs Quantitation reports for all ICVs
Continuing Calibration Data (both columns)	Summary of %Ds and calibration factors Chromatograms for all continuing calibration standards Quantitation reports for all continuing calibration standards Concentrations of standards used must be clearly presented
Sample Results (both columns)	Chromatograms for all sample analyses, reanalyses, and dilutions



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DELIVERABLE REQUIREMENTS FOR DATA AUDITS WSC-CAM-V C (Chlorinated Herbicides by GC/ECD)	
	Quantitation reports for all sample analyses, reanalyses, and dilutions Percent solids results Summary of results, including reporting limits for each sample Date of analysis
Method Blank Results (both columns)	Chromatograms for all method blanks Quantitation reports for all method blanks Summary of results, including reporting limits Summary of how method blank was prepared in solid and aqueous matrices, as appropriate
LCS/LCS Duplicate Results (both columns)	Chromatograms for all LCS and LCS Duplicates Quantitation reports for all LCS and LCS Duplicates Summary of results, including concentrations detected, concentrations spiked, percent recoveries and RPDs Summary of how LCS/LCS Duplicates were prepared in solid and aqueous matrices, as appropriate
MS/MSD Results (if performed) (both columns)	Chromatograms for all MS/MSDs Quantitation reports for all MS/MSDs Summary of results, including unspiked sample concentrations, concentrations detected, concentrations spiked, percent recoveries and RPDs Summary of how MS/MSDs were prepared in solid and aqueous matrices, as appropriate
QC Summaries (both columns)	Surrogate recoveries Internal standard performance Retention time windows Dual column RPDs
Other Information	Demonstration that ICV prepared from second source standard
Quantitation reports must exhibit peak area counts or peak heights, as appropriate, of target compounds, internal standards, and surrogates. ¹ Must clearly indicate sample weights or volumes, final extract volumes, extraction method used, extraction times where appropriate for the method, etc.	